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Metabolic heat regenerated Temperature Swing Adsorption for CO₂ & Heat Removal/Rejection in a Martian PLSS

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ABSTRACT

Two of the fundamental problems facing the development of a Portable Life Support System (PLSS) for use on Mars, are (i) heat rejection (because traditional technologies use sublimation of water, which wastes a scarce resource and contaminates the premises), and (ii) rejection of carbon dioxide (CO₂) in an environment with a CO₂ partial pressure (ppCO₂) of 0.4-0.9 kPa. Patent-pending *Metabolic heat regenerated Temperature Swing Adsorption* (MTSA) technology is being developed to address both these challenges. The technology utilizes an adsorbent that when cooled with liquid CO₂ to near sublimation temperatures (~195K) removes metabolically-produced CO₂ in the ventilation loop. Once fully loaded, the adsorbent is then warmed externally by the ventilation loop (~300K), rejecting the captured CO₂ to Mars ambient. Two beds are used to provide a continuous cycle of CO₂ removal/rejection as well as facilitate heat exchange out of the ventilation loop. Any cryogenic fluid can be used in the application; however, since CO₂ is readily available on Mars and can be easily produced and stored on the Martian surface, the solution is rather elegant and less complicated when employing liquid CO₂. As some metabolic heat will need to be rejected anyway, finding a practical use for metabolic heat is also an overall benefit to the PLSS. To investigate the feasibility of the technology, a series of experiments were conducted which lead to the selection and partial characterization of an appropriate adsorbent. The Molsiv Adsorbents 13X 8x12 (also known as NaX zeolite) successfully removed CO₂ from a simulated ventilation loop at the prescribed temperature swing anticipated during PLSS operating conditions on Mars using a cryogenic fluid. Thermal conductivity of the adsorbent was also measured to eventually aid in a demonstrator design of the technology. These results provide no show stoppers to the development of MTSA technology and allow its development to focus on other design challenges as listed in the conclusions section of this paper.

INTRODUCTION

Portable Life Support Systems (PLSS) for use on Mars need to be redesigned to address a set of challenges different than those posed during space extravehicular activity (EVA) or on the lunar surface. Removal of metabolically-produced carbon dioxide (CO₂) from an astronaut's air supply (ventilation loop) is typically accomplished with lithium hydroxide (LiOH) canisters. Not being regenerable, these canisters limit operations, provide a logistical volume and mass issue, and are expensive to replenish. Typical regenerable technologies use pressure swing to collect and then reject the metabolically-produced CO₂. However, the appropriate lower pressure is not available on Mars since its environment has a CO₂ partial pressure (ppCO₂) of 0.4-0.9 kilopascals (kPa).

Another challenge faced by a PLSS on Mars is thermal control of the astronaut. Heat rejection is a prevalent need and often accomplished through sublimation of water. The sublimated water is rejected from the PLSS into and onto the surrounding environment, not only wasting a critical resource but also contaminating the premises and hence any scientific return.

Patent pending *Metabolic heat regenerated Temperature Swing Adsorption* (MTSA) technology is being proposed as a solution to these Martian challenges. Traditional temperature swing adsorption (TSA) technologies collect CO₂ at ambient temperatures and then are heated up to reject the CO₂ and regenerate. This heat up "cost" has lead most to believe that TSA is impractical for PLSS applications. However, it could be possible to use TSA in a "cooler" range, where the temperature of the heat source for regeneration does not have to be as high. Since heat rejection is a prevalent challenge in PLSS design, using metabolic heat for adsorbent regeneration would be a very elegant solution to both thermal and CO₂ management.

To achieve a significant loading on the adsorbent using only metabolic heat, the adsorbent will have to be cooled significantly. If an adsorbent exists with loading characteristics that satisfies these operating conditions, any cryogenic fluid can be used. However, there is an adequate coolant readily available from Martian resources that can be collected and stored for relatively low power and minimal infrastructure: liquid CO₂.

BACKGROUND

The MTSA technology is described in detail elsewhere [Error! Reference source not found.]. Briefly, metabolically-produced CO₂ is removed from a pre-conditioned cooled ventilation loop (40kPa) of a PLSS using an adsorbent such as Molsiv Adsorbents 13X 8x12 (also known as NaX zeolite). To increase the capacity of the adsorbent, it is cooled using either a cryogenic fluid or expanded liquid CO₂ (LCO₂).

Using LCO₂ on Mars has many advantages. 1) It can be produced on Mars with relatively minimal infrastructure and low energy costs [Error! Reference source not found.]. 2) After the LCO₂ is used by the MTSA (now a gas), it still has cooling capacity to aid in additional heat rejection, e.g., via a heat exchanger with a liquid cooling garment. 3) Once all the cooling capacity of the LCO₂ is consumed, the used gaseous LCO₂ is safely expelled to the Martian atmosphere where it does not contaminate the surrounding environment since Martian atmosphere is 95% CO₂.

Once the adsorbent is full, it rejects the collected metabolically-produced CO₂ to Mars ambient (0.8kPa) when it is warmed by a heat exchanger with the gas stream of the unprocessed warm ventilation loop. The effective temperature swing is somewhere between the CO₂ sublimation temperature (~195K) and the ventilation loop temperature (~300K). Continuous removal of metabolically-produced CO₂ is achieved by cycling between two beds.

Each bed will perform the same loading cycle as shown in Figure 1 and Figure 2. Figure 1 demonstrates how each bed works in adsorb and desorb modes to ensure continuous CO₂ removal. Figure 2 demonstrates how the CO₂ loading changes with temperature, pressure and gas composition. To demonstrate the different conditions in which the adsorbent will operate, Bed B is used as an example starting with Point 1.

Point 1: The adsorbent is warm (T_{desorb}), unloaded and exposed to Mars ambient (CO₂ at 0.8 kPa). The outlet valve of bed B is then closed and bed B is cooled to T_{adsorb} .

Point 2: The adsorbent is cold (T_{adsorb}) and still unloaded. The adsorbent is then exposed to the ventilation loop (O₂/CO₂ at 40 kPa). The adsorbent is continuously cooled to counter the heat of adsorption of CO₂ onto the adsorbent and be maintained at T_{adsorb} .

Point 3: The adsorbent is cold (T_{adsorb}), loaded and exposed to the ventilation loop (O₂/CO₂ at 40 kPa). The adsorbent is then exposed to Mars ambient in preparation for desorb mode.

Point 4: The adsorbent is still cold (T_{adsorb}), partially loaded and exposed to Mars ambient (CO₂ at 0.8 kPa). It is only partially unloaded due to the pressure drop when switching the ventilation loop to bed A and exposing the adsorbent in bed B to Mars ambient.

Path 5: The adsorbent is warmed to T_{desorb} while exposed to Mars ambient. Once the adsorbent is unloaded, the cycle is repeated.

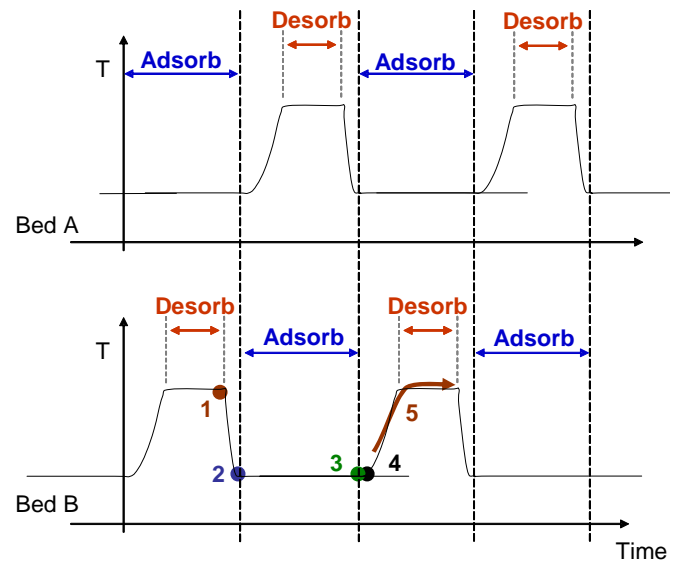


Figure 1: Loading Cycles

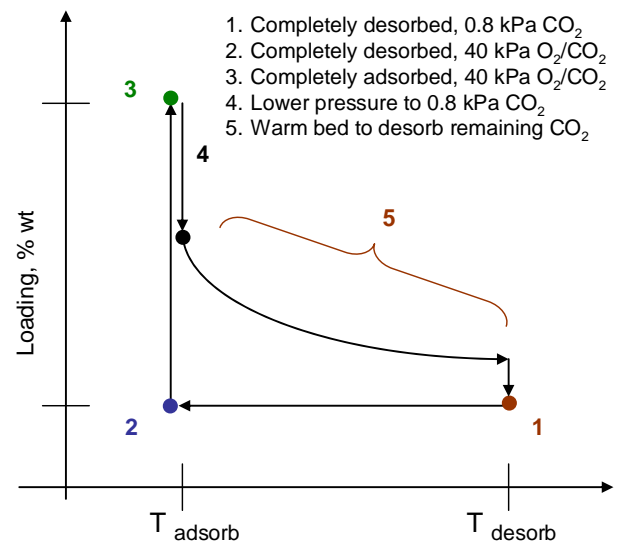


Figure 2: Loading Changes vs Temperature

Two feasibility issues of the MTSA concept were identified: 1) locating an adsorbent conducive to MTSA operating conditions and 2) understanding how it performs (both loading capability and thermally) to design the best MTSA subsystem.

Isobaric loading data (0.8kPa) over the temperature range of interest (210 – 280K) for an adsorbent called NaX was located [2]. Preliminary sizing calculations using this data suggest NaX is sufficient to meet the needs of a MTSA for a Martian PLSS [Error! Reference source not found.]. However, no data for the loading condition (210K at 40kPa) has been located.

Additionally, thermal conductivity of adsorbents is not widely published, if at all. It may be a challenge to transfer metabolic heat from the ventilation loop to the adsorbent if the adsorbent thermal conductivity is low. Only after the thermal conductivity is known can it be attempted to correctly design an MTSA subsystem.

For these reasons, two feasibility experiments were conducted: one to assess the CO₂ loading of NaX over the temperature and pressure range of interest and another to assess the thermal conductivity of the adsorbent. The design, testing apparatus and results of these two tests are presented below.

FEASIBILITY EXPERIMENTS

TEST SET UP

Both experiments described here used the same test bed. A schematic of the test bed is shown in Figure 3. A dry ventilation loop is simulated with O₂ and CO₂ gas at 5.1 kg/hr and 0.1 kg/hr, respectively, using Alicat flow controllers. A backpressure controller (Alicat) and oxygen-rated vacuum pump maintain the ventilation loop pressure at 40kPa. A temperature chamber cools the test article to 210K. A copper tube is used as a heat exchanger prior to the test article to ensure the simulated ventilation loop achieves the desired temperature. Plug type thermocouples (Omega, TC-T-NPT-G-72) verify the ventilation loop entry and exit temperatures to the test article. The ventilation loop passes through the test article, where CO₂ is removed. A CO₂ detector (Analox Model 5S MKII) and sample pump monitor for CO₂ breakthrough down stream of the chamber indicating the adsorbent bed inside the test article is “full”. During desorb mode, the simulated dry ventilation loop is turned off. The inlet to the test article is closed and the outlet is exposed to simulated Mars ambient (100% CO₂ at 0.8kPa) using the same vacuum pump and back pressure regulator as used for the simulated ventilation loop. A filter is down stream of the test article to catch any particulates from the adsorbent.

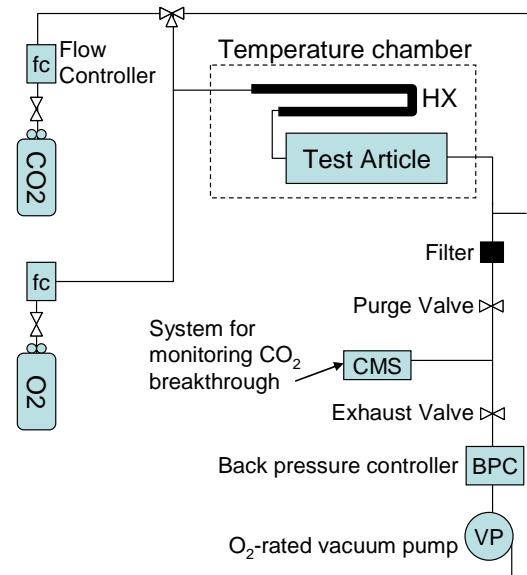


Figure 3: Schematic of test bed

CO₂ LOADING EXPERIMENTS

Objectives

After an extensive literature search for candidate adsorbent materials that would operate well under the required operating conditions, a final down select to two was made. NaX zeolite and activated carbon were chosen primarily due to promising CO₂ loading data [2],[3]. In the end, NaX was chosen for characterization because a greater quantity of CO₂ data were found with some matching the desorb condition exactly, there is significant flight history (i.e. NaX is used in the Carbon Dioxide Removal Assembly (CDRA) on the International Space Station (ISS)), and a supplier was easily located. In addition to selecting an adsorbent for study, the literature search also yielded other applicable information to the system. Work done on similar systems had issues when trying to run low flow rates of an inert gas along with CO₂ where the inert gas would build up and cause a blockage preventing good CO₂ adsorption. Another problematic area that was cautioned against was the channeling of CO₂, especially if bed length was not large compared to diameter. From operation of the CDRA on ISS, particulate zeolite could also become a problem as it could contaminate downstream components restricting their proper operation. However, through discussions with Honeywell¹, the causes and mitigation methods of this phenomenon are well understood and therefore addressable in an eventual flight design. Finally, more recent work using zeolite on a substrate seemed to promise several advantages to a packed bed including improved loading, decreased pressure drop, and increased heat transfer. This last item, while intriguing, was viewed as a direction for possible future work, but

¹ Private discussions with the Honeywell CDRA Project Lead, AIAA Aerospace Conference, Reno, NV January 2005.

the initial characterization would continue with a packed bed in large part due to time constraints.

We performed a series of CO₂ loading experiments using NaX Zeolite (UOP LLC). The primary objective of these tests was to obtain data that can be used to assess the ability of NaX to meet the required CO₂ loading/unloading under MTSA operating conditions (temperature and pressure) for a PLSS. The following specific objectives were achieved:

1. Measure the CO₂ loading on an adsorbent at 40kPa total pressure, 210K, and PLSS ventilation loop O₂ and CO₂ mass flow rates (5.1 and 0.1 kg/hr, respectively).
2. Measure the CO₂ unloading at 280K and 0.8kPa (6 torr) 100% CO₂ (simulated Mars ambient).
3. Measure the CO₂ unloading at 266K and 0.8kPa (6 torr) 100% CO₂ (simulated Mars ambient).

Test Article Description

In order to best quantify a given amount of adsorbent material during the experiment, the diameter of the bed needs to be minimized. This causes an even CO₂ loading front that passes through the bed, maximizing the usage of the adsorbent before CO₂ breakthrough. Small diameters cause an increase in mass (added weight due to a longer casing) and thus it should be noted that there would be a trade on MTSA mass versus efficient use of the adsorbent.

The test article is a 61-1/2" aluminum tube (Part Number 89965K58, McMaster-Carr) of 0.555" inner diameter and 5/8" outer diameter. On the inlet and outlet, 18x18-mesh stainless steel screens (McMaster-Carr) are inserted 3 inches into the tube to hold the adsorbent material in place. Adsorbent (150.4 g, UOP, 1/16 inch dia. pellets) fills the space between the two screens. To fit in the temperature chamber, the tube is looped two and half times. T-type plug probe thermocouples (PN TC-T-NPT-G-72, Omega Engineering, Inc.) are inserted at each end of the tube via t-fittings to monitor the temperature of the inlet and outlet gasses. A copper tube (Part Number 8955K12, McMaster-Carr) acts as a heat exchanger upstream of the test article. A picture of the assembly is shown in Figure 4 before the Swagelok® fittings were replaced with flare fittings to overcome leaks incurred by thermal cycling from adsorbent conditioning (450 K to loading 210 K).

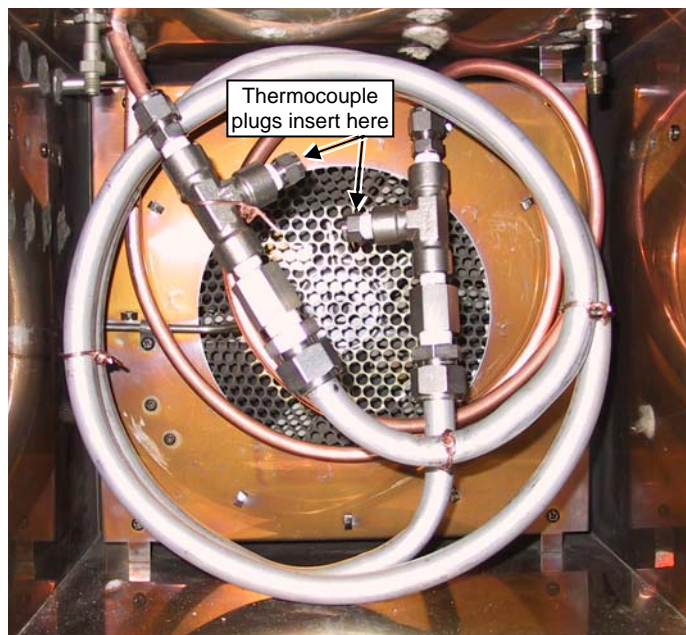


Figure 4: CO₂ loading test article with upstream copper heat exchanger. Swagelok® fittings were eventually replaced with 37° flare fitting to minimize leaks during extreme thermal cycling.

Results

The mass of CO₂ loaded per the mass of adsorbent (%wt) was calculated assuming a fixed CO₂ mass flow rate over a time span from the adsorbent's first exposure to the ventilation loop to CO₂ breakthrough. CO₂ in the lines upstream of the test article and downstream between the test article and CO₂ detection system were estimated and subtracted from the loaded CO₂ mass estimates. Outside of any test anomalies, the experiments were terminated when the last cycles produced loadings within 10% of each other. An example set of data is shown in Table 1.

Table 1: Total CO₂ Loaded for Adsorb T = 210K and Adsorb Total P = 40kPa, Desorb T = 280K and Desorb Total P = 0.8kPa, CO₂ Desorb Time = 7 mins

Cycle	Total CO ₂ loaded (g)	Overall CO ₂ loading (%wt)
1	22.0	14.6
2	17.4	11.6
3	16.6	11.0
4	15.9	10.6
5	16.9	11.2

The amounts varied depending on loading conditions and time. The greatest amount of CO₂ adsorbed over a cycle was achieved with the longer desorb time and higher desorb temperature (assuming a fixed set of loading conditions). Increasing desorb time allows the adsorbent to reject more CO₂ at the prescribed temperature and pressure. Increasing the desorb temperature forces more CO₂ off of the adsorbent.

For our testing, a maximum average load of 14.7%wt was achieved over a cycle from 210 K, 40 kPa to 280 K, 0.8 kPa where unloading was performed over 25 minutes.

Of interest to the MTSA application is the loading over a cycle, which is time dependent. Thus the desorb time was decreased for subsequent tests to assess the sensitivity to desorb time. In other words, reducing the desorb time does not let the adsorbent achieve equilibrium at the desorb conditions. This results in the adsorbent holding more CO₂ and reducing the overall loading capability. To investigate this phenomenon, the desorb time was reduced from 25 minutes to 7 minutes.

An average loading of 11.1%wt was achieved over a cycle from 210 K, 40 kPa to 280 K, 0.8 kPa where unloading was shortened to 7 minutes. To evaluate a “worst case” scenario where target desorb temperature is not achieved and target loading time is shortened, another experiment was performed. An average loading of 9.1%wt was achieved over a cycle from 210 K, 40 kPa to 266 K, 0.8 kPa where unloading was performed again over 7 minutes.

Figure 5 summarizes the CO₂ loading percent weight calculated for different operating conditions. A detailed uncertainty analysis results in .03 – 0.33% (% wt fraction) uncertainty indicate testing methods were reasonable.

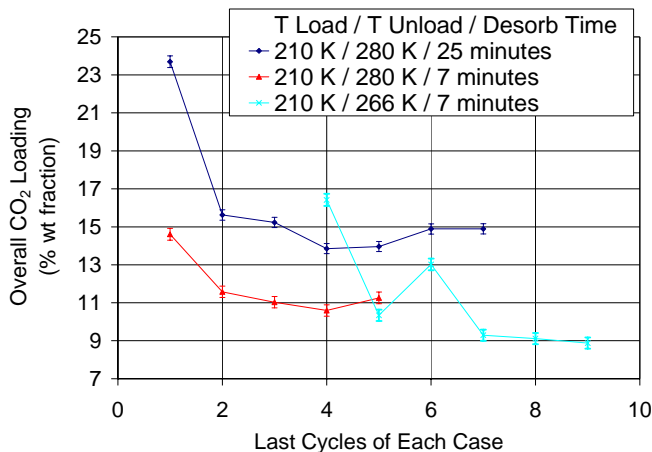


Figure 5: Overall CO₂ loading in the end cycles of each loading.

ADSORBENT THERMAL CONDUCTIVITY EXPERIMENTS

Objectives

A second test was developed to characterize the thermal conductivity of NaX Zeolite so that a proper model could be built to design an MTSA capable of being warmed sufficiently with a moist ventilation loop alone.

The adsorbent operates in various temperatures, pressures, loadings and in the presence of various gas compositions. All these conditions affect the heat transfer capability of the adsorbent bed. Referring to Figure 2, point 4 and path 5 are of variable loading depending upon how the operating conditions change (temperature and pressure). Points 1, 2 and 3 can be achieved in steady state in the lab and thus make for good test cases in which to measure the thermal conductivity. These measurements should bound the performance of the adsorbent.

With this in mind, the following objectives apply to this test:

1. Assess the thermal conductivity of the unloaded adsorbent material in the sample space of the test article at the desorb temperature (i.e. at point 1 of Figure 2):
 - a. Adsorbent: Unloaded
 - b. Temperature Chamber: 280 K
 - c. Gas: no flow, outlet exposed to CO₂ at 0.8 kPa
2. Assess the thermal conductivity of the unloaded adsorbent material just before loading begins (i.e. at point 2 of Figure 2):
 - a. Adsorbent: Unloaded
 - b. Temperature Chamber: 210 K
 - c. Gas: inlet and outlet closed, internal pressure 0.8 kPa
3. Assess the thermal conductivity of the adsorbent material after loading at the adsorb temperature (i.e. at point 3 of Figure 2):
 - a. Adsorbent: Loaded
 - b. Temperature Chamber: 210 K
 - c. Gas: ventilation loop O₂/CO₂ at 40 kPa

Test Article Design and Description

Typical thermal conductivity experiments use a longitudinal section of the sample material and a reference material that both transmit one dimensional (1-D) heat transfer. However, this type of test set up would not suffice since it was anticipated that the thermal conductivity of the adsorbent would be so low that heat leak to the surrounding environment in the lateral direction would be on the same order as that traveling longitudinally, violating the 1-D assumption needed to calculate the thermal conductivity of the adsorbent with respect to a reference material.

In order to remove the large areas for lateral heat transfer, a polar system was considered. An apparatus of this type has a heated cylindrical center which conducts evenly through the surrounding test media to a

surrounding sink. This test is very similar to the typical longitudinal test in that the heat transfer is one dimensional but now the heat transfer is along the radial coordinate rather than the longitudinal coordinate. This radial heat transfer is described by the equation:

$$q_r = 2\pi Lk \frac{\Delta T}{\ln(r_2 / r_1)} \quad (1)$$

Where q_r is heat flow in Watts at an arbitrary radius, L is the length of the article in meters, r_2 is the outer radius of interest in meters, and r_1 is the inner radius of interest in meters.

The entire test article is made out of Teflon, which serves as a low thermal conductivity reference material. The test article is comprised of an annulus that is created by a Teflon cylinder shell and an inner solid Teflon cylinder core (see Figure 6). The core houses a heater cartridge as well as ten thermocouples. The heater cartridge is inserted into the center of the top of the core and heats the core along its longitudinal axis. Four sets of two thermocouples (TCs) are strategically placed to measure the temperature gradient radially outward (see Figure 8, TC 1 & 2, TC 3 & 4, TC 5 & 6, TC 7 & 9). These thermocouples reach a uniform depth half way down the cylinder (see Figure 7). In addition, two more thermocouples are placed at 45° off the radial measurements (TC 8 & 10) at a depth shallower than the other thermocouples. These additional thermocouples check for uniformity of temperature along the length of the core. Four external thermocouples on the shell align with the radial temperature measurements. Temperatures measured here are used to back out the thermal conductivity of the material in the sample space.

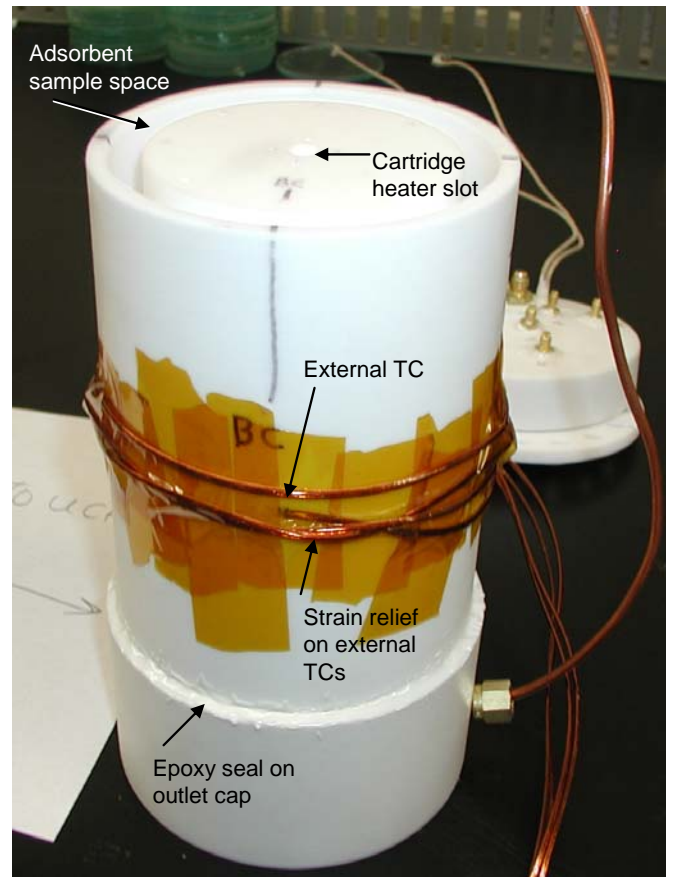


Figure 6: Annulus created for sample space (retention disk and inlet cap not shown)

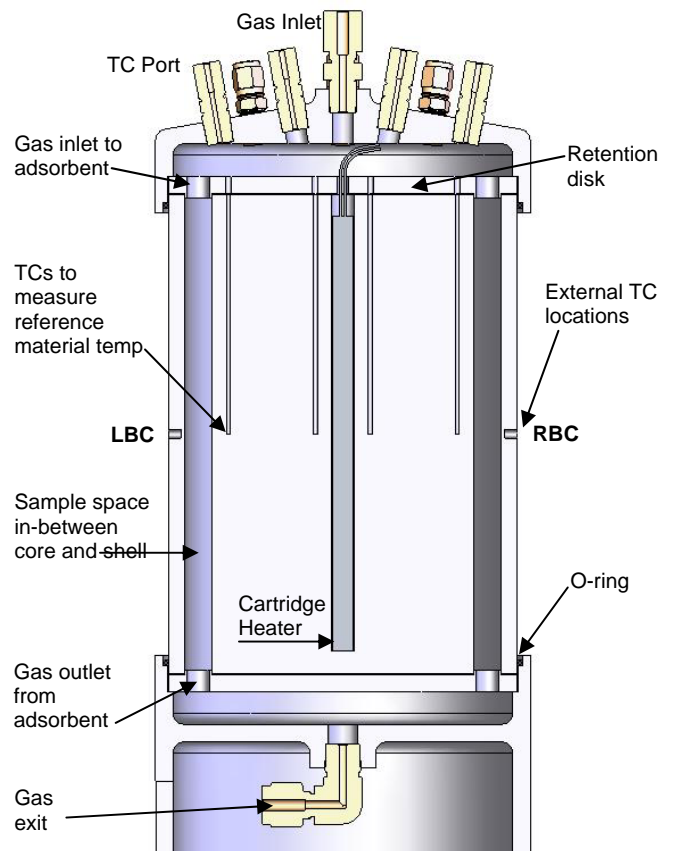


Figure 7: Cross Section of Test Article

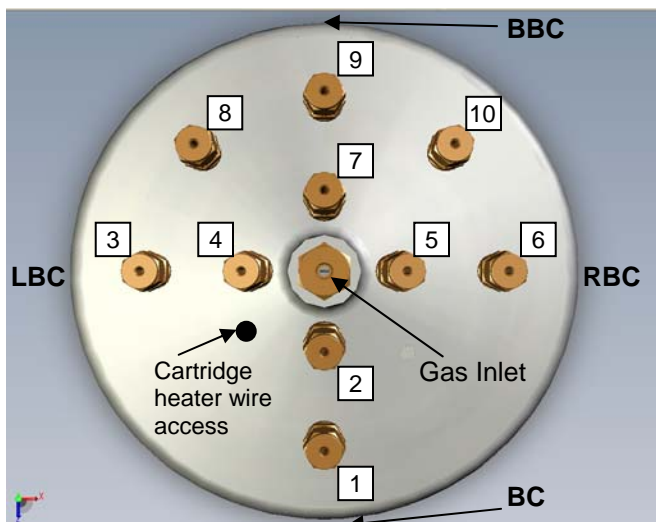


Figure 8: Thermocouple Arrangement on top of inlet cap (1 – 6 and 7 & 9 measure the radial temperature gradient half way along the core; 8 & 10 check uniformity of that temperature gradient at shallower depths; BC, RBC, BBC, LBC are labeled to indicate approximate positions of external temperature measurements on the shell.)

The annulus created by the core and the shell is the sample space. A keyed sorbent retention disk is placed on top of the assembly to enclose the sample space. An inlet cap is placed on top. The inlet cap is fitted with Swagelok fittings to allow the thermocouples and cartridge heater wire access (see Figure 9). The key in the retention disk ensures the ports are aligned with the TCs in the core.

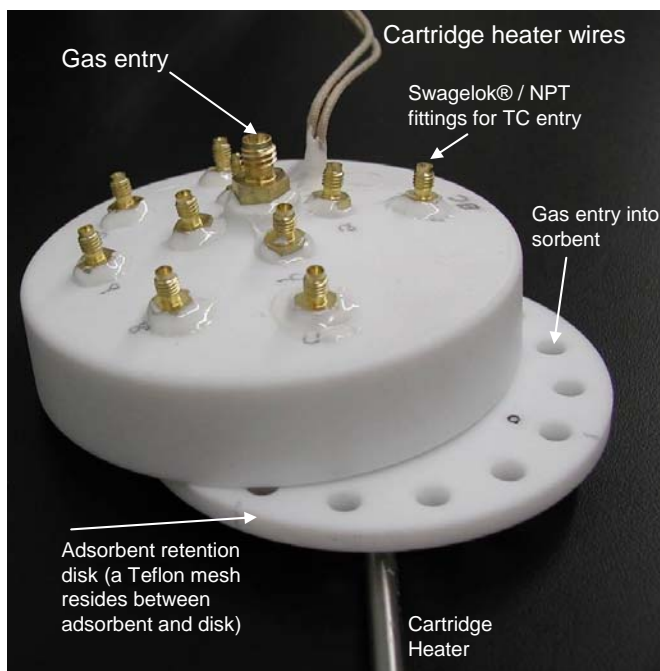


Figure 9: Inlet cap and retention disk

In order to seal the space around the thermocouples, the thermocouples are wrapped in heat shrink tubing and secured with Teflon ferrules in Swagelok® fittings. The

Swagelok® fittings were secured with a vacuum rated epoxy (Torr seal). Seals between the caps and the test shell were attempted using Teflon O-rings; however, in the end, to achieve a leak tight seal under 0.8 kPa conditions, the same vacuum rated epoxy had to be used. To reduce the heat transfer between the test core and the caps, Teflon mesh screens are inserted to increase contact resistance.

Results

The test cases achieved are listed in Table 2. All temperature data was plotted versus time to assess if the system achieved steady state. For each case, analysis of the core reference material temperatures indicate that isotherms were successfully achieved around the heater cartridge, with the hottest isotherm near the heater cartridge and the coldest at the outer edge of the reference core material. Furthermore, similar temperatures along the longitudinal section of the reference core material indicate 1-D radial heat flow was achieved in the mid-section of the test article.

Table 2: Description of Test Cases

Case (objective)	T (K)	Power (W)	P (kPa)	Achieve Steady State?
Warm Low Power (1)	280	1.9	0.8	Yes
Warm High Power (1)	280	2.8	0.8	Yes
Cold Low Power (2)	210	1.9	0.8	No

The warm, low power case and the warm, high power case achieved steady state (see Figure 10 for example). The cold, low power case did not completely achieve steady state due to depletion of the liquid nitrogen (LN₂) used to maintain the cold temperature (see Figure 11). However, calculations were performed on that data set in the hope of obtaining more information since the changes were occurring slowly. A high target CO₂ loading test case (objective 3) was not accomplished due to the test article seal being compromised when a piece of adsorbent plugged the exhaust line.

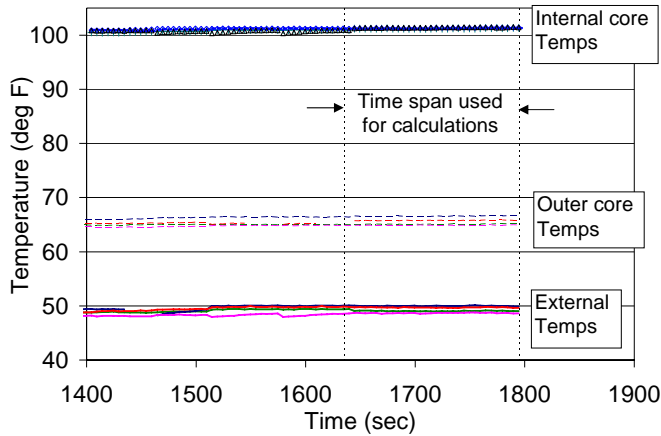


Figure 10: Example data from the warm, high power case (30 August 2006, Time 0 = 16:26 PSDT)

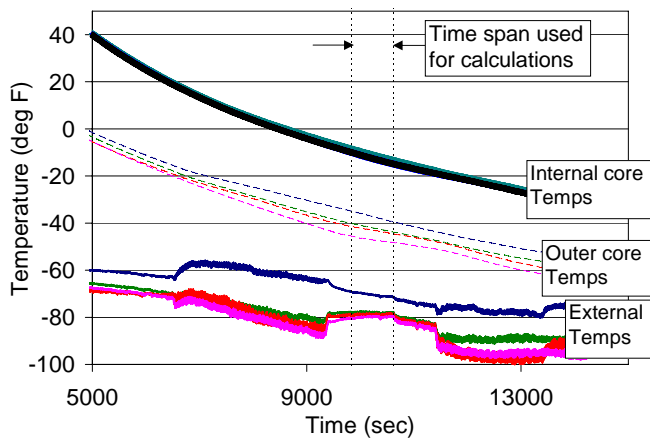


Figure 11: Example data from the cold, low power case. Noise in external temps is due to LN₂ tank depletion. (30 August 2006, Time 0 = 18:55 PSDT)

ANALYSIS

Adsorbent Model Sizing

An EXCEL model was completed to size the adsorbent bed using readily available adsorbent data as retrieved through the literature search and via vendors. Specifically, NaX data published by Finn et al. [2] and the vendor UOP were used.

The model assumes two beds for cycling between adsorb and desorb modes such that continuous CO₂ removal can be maintained. The adsorbent bed casing is modeled as well. Additional assumptions include a 1 inch end gap for glass packing, and end hardware was not included (i.e. fittings). The resulting amount of sorbent required for the experiment described above is 150.4 mg. This assumes the maximum loading of a PLSS (50 mg/s) loaded for 10 minutes. Specific model parameters are presented in Table 3.

Table 3: Adsorbent bed sizing model parameters

Variable Description	Value
Collection Time:	10.0 min
CO ₂ Flow Rate ¹ :	1527.3 sccm
	50.0 mg/s
Desired CO ₂ Collection per bed:	0.03 kg
Number of Units:	2.0
Number of cycles/unit/EVA ² :	24.0
Gap, sorbent to wall:	0.5 cm
End gap:	2.5 cm
Wall thickness:	0.1 cm
Canister Material:	Aluminum
Canister Density:	2.70 kg/L
Canister Cp:	0.90 kJ/kg-K
Sorbent:	NaX Zeolite
Sorbent Density:	0.7 g/ml
Cp for Sorbent:	0.9 kJ/kg-K
Heat of adsorption:	41 kJ/mol
Inlet Pressure of Ventilation Loop:	40.0 kPa
Inlet Temperature of Ventilation Loop:	232 K
CO ₂ Loading at Input ³ :	25.7 %wt
Desired Output Temperature:	280 K
Desired Output Pressure (6 torr):	0.8 kPa
CO ₂ Loading at Desired Output [10]:	5.79%wt
Sorbent Mass/Unit:	150.4
Required Volume/Unit:	0.2

¹ standard cm³/min where Tstp=273K, Pstp=101.325kPa

² Assumes 8 hour EVA

³ Only have 232 K data for low temperature from UOP

Results from the model are shown in Figure 12 labeled "Fully equilibrated data (UOP)". Several scenarios of system mass are also presented assuming the loading as achieved in the experiments using the bed design of the test article. The worst case scenario presents the bed size should the adsorbent not be given enough time to desorb and not be given enough time to achieve 280K. The next two cases demonstrate that longer desorb times can cause a bigger overall CO₂ loading. Compared to the data available from UOP, a bigger percentage of CO₂ loading could probably have been achieved if greater than 25 minutes were allotted for the desorb time. This does not imply an effective design needs greater than 25 minutes to realize the greatest overall CO₂ loading. The experiment used a long, skinny tube using only the pressure generated by the desorbing CO₂ to reject the CO₂ to the simulated Martian atmosphere. Elution paths, amount of adsorbent, effective use of the adsorbent and cycle time will have to all be considered when designing a final bed to meet PLSS requirements within MTSA operating conditions.

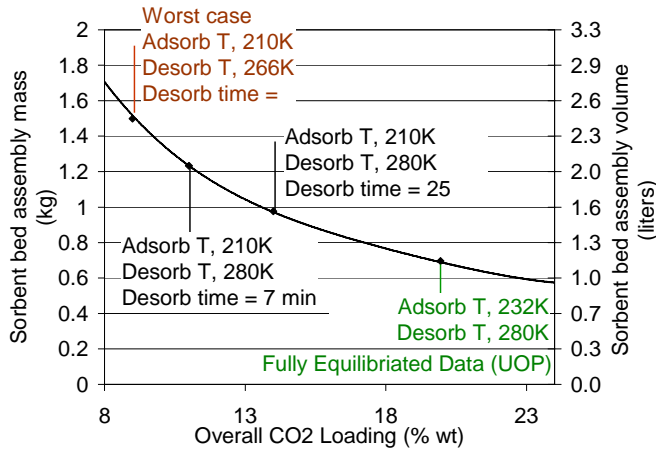


Figure 12: Sizing of adsorbent bed assembly based on loading experiments and compared to fully equilibrated data as provided by UOP

Thermal Conductivity Calculations

The thermal conductivity of the adsorbent was calculated assuming the heat transfer out of the test core was conserved in the radial direction (1-D heat flow). To assess temperature uniformity in the longitudinal direction and thus adequacy of this assumption, two temperatures were measured along the outer portion of the test core material *above* the plane that contained the thermocouples used for the thermal conductivity calculations. These thermocouples (TC 8 and 10) were compared to the other outer core temperatures (TC 1, 3, 6 and 9) and confirmed the uniformity assumption.

Once uniformity was confirmed, the heat transfer from T_a to T_b (the inner to outer most thermocouples in the core reference material, respectively) could be equated to the heat transfer from the inner edge of the adsorbent ($T_{b'}$) to the outer edge of the adsorbent (T_c). Applying equation 1 above for radial heat transfer, canceling $2\pi L$ and rearranging, the following equation was used to calculate the thermal conductivity of the adsorbent:

$$k_s = k_R \frac{\ln(r_c / r_{b'}) \Delta T_{a-b}}{\ln(r_b / r_a) \Delta T_{b'-c}} \quad (2)$$

Where k_s is the thermal conductivity of the sample adsorbent material, k_R is the thermal conductivity of the reference core material (Teflon, 0.2 W/m-K), $r_{b'}$ is the distance of the inner edge of the adsorbent (0.038 m), r_c is the distance of the outer radius of the entire test article (0.046 m), and ΔT_{x-y} is the temperature difference between thermocouple T_x and thermocouple T_y in Kelvin. The temperature at $r_{b'}$ is calculated by solving the temperature distribution in the radial direction:

$$T(r_{b'}) = \frac{(T_b - T_a)}{\ln(r_b / r_a)} \ln(r_{b'} / r_a) + T_a \quad (3)$$

For each case there are potentially four sets of data that can be used to calculate the thermal conductivity of the adsorbent. Each valid calculation is presented in Table 4 and then averaged for each case. Note that the results for the cold, low power case are using data that was not fully in steady state but changes were occurring slowly enough that we think the calculations are indicative of trends. An uncertainty analysis was performed according to standard equations of error propagation. The biggest contributor to uncertainty was the thermocouples of Tolerance Class 1, which gives them a precision of $\pm 1^\circ \text{C}$.

Table 4: Thermal conductivity calculations for NaX at various conditions

Case	Thermal Conductivity (W/m-K) ¹				
	k 2-1	k 4-3	k 7-9	k 5-6	Avg.
Warm, low power	0.036	0.043	--	0.056	0.045 \pm 0.011
warm, high power	0.076	0.069	0.067	0.075	0.072 \pm 0.019
cold, low power ²	0.024	0.023	0.025	0.030	0.025 \pm 0.003

¹ Given in terms of the thermocouple pair used to generate the calculations (i.e. k 2-1 used TC 2 & TC 1).

² Steady state not fully achieved for this set of calculations so use with caution.

CONCLUSIONS

Loading experiments were performed to demonstrate that NaX adsorbent performs well under MTSA operating conditions, achieving 11 – 15%wt CO₂ loading depending upon desorb time for the given test article design. The conclusion is that overall loading is driven by desorb time. This is not a limitation of the technology but rather a design constraint. Decreasing overall CO₂ loading by decreasing the desorb time indicates an inefficient use of the adsorbent mass. In fact, for a given set of conditions the percent by weight loading should remain the same. Decreasing the desorb time just means we didn't allow the sample enough time to desorb. We have thus learned, that for shorter desorb times we should decrease the size of the bed such that the adsorbent is used most efficiently. To optimize the bed size, more experiments should be run to learn what the minimum desorb time is to achieve equilibrium conditions within the bed and for a given bed design.

Thermal conductivity of a packed bed of NaX zeolite was measured. At 280 K in a 0.8 kPa CO₂ environment, the values ranged from 0.036 to 0.076 \pm 0.019 W/m-K. These were determined in the presence of low and high power settings, and were higher by 60% in the high power case. At 210K in a 0.8 kPa CO₂ environment, the thermal conductivity ranged from 0.023 – 0.030 \pm 0.003 W/m-K. Not enough experiments were run to gain

confidence in these numbers; however, we feel they are an acceptable order of representative magnitude. Given that the challenge of MTSA technology is heating the adsorbent using metabolic heat from the ventilation loop, future modeling should assume conservative low values on the order of:

$$k_{\text{adsorbent}} (T=280\text{K}) = 0.035 \text{ W/m-K}$$

$$k_{\text{adsorbent}} (T=210\text{K}) = 0.025 \text{ W/m-K}$$

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